High-resolution X-Ray Diffraction Studies of Highly Curved GaN Layers Prepared by Hydride Vapor Phase Epitaxy

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ABSTRACT

In addition to dislocations, the wafer curvature can also affect the broadening of x-ray rocking curve (XRC) peaks and it may deteriorate the accuracy of the tilt and twist angle measurements. In this paper, the radial-distribution of curvature and the effects of wafer curvature on XRC of highly curved (the radius curvature of *r* less than 2.5 m) GaN layers were studied. Curvature-related effects both on symmetric geometry and on the (102) skew symmetric geometry were studied by the use of adjustable beam slits and 'antis.slit' placed before the sample and before the detector, respectively. The acceptable approaches to minimize the curvature-related effects in determination of a reliable tilt or twist angle were proposed. It is found that the curvature-related effects can be eliminated by the use of the methods we proposed. The dislocation densities obtained by high resolution x-ray diffraction (HRXRD) are fairly consistent with that obtained by cathodoluminescence (CL) and atomic force microscope (AFM).

Keywords: A1.Tilt and Twist; A1.XRC; A1.HRXRD; A1.Curvature; B2.GaN

1. INTRODUCTION

Gallium nitride (GaN) and its alloys with indium or aluminum nitride have attracted significant attention in recent years due to the successful development of visible and ultraviolet light emitting diodes [1], blue/violet laser diodes [2], and high-power electronic devices [3] based on this materials system. GaN layers are generally grown on substrates such as sapphire, silicon, or SiC [4], all of which have lattice parameters and thermal expansion coefficients that markedly

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differ from those of GaN. Therefor the GaN layers are generally bent and possess of high threading dislocation densities. Hydride vapor phase epitaxy (HVPE) technology has been developed to grow thick or free-standing GaN layers to decrease the threading dislocation density (TDD). The TDDs with 10^7~10^8 cm^2 are now common for GaN layers prepared by HVPE [5] and other improved crystal growth methods [6-7]. Bur such layers are often exceptionally highly curved. Radiuses of curvature of 3 m or less are familiar especially for the HVPE-grown thick GaN layers.

In the high resolution x-ray diffraction (HRXRD) analysis of GaN layer, the full-width at half-maximum (FWHM) of x-ray rocking curve (XRC) is often used as a parameter to determine the tilt and twist angle [8], whose square is approximately proportional to the screw and edge type dislocation density respectively [9-10]. In addition to dislocation and grain size, the wafer curvature also affects the broadening of XRC peaks [11-13]. The change of XRC-FWHM with the bending has been observed in curved layer [12-13] and it may deteriorate the accuracy of the angle measurements. The curvature-related effects and the way to eliminate the effects from XRC with an analyzer have been discussed by Liu [14]. In this paper, we discussed the curvature-related effects and the way to eliminate the effects from XRC without an analyzer.

2. EXPERIMENTS

Four 2 inch (0001)-oriented crack-free GaN samples A, B, C and D with thicknesses 12 μm, 20 μm, 38 μm and 300 μm were grown on 2 μm MOCVD-grown GaN/sapphire templates in an HVPE system with a horizontal quartz reactor—HCl/metal Ga, ammoniaand N_2/H_2 mixture were used as gallium source, nitrogen source and carrier gas, respectively. The growth rate was typically about 70 μm /h. Another two MOCVD grown GaN layers with thicknesses 200nm and 600 nm were used for contrastive study.

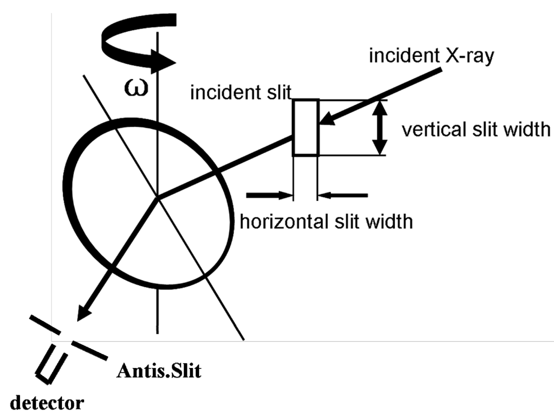

Fig. 1. Schematic diagram of the X-ray measurement system in this work.

The HRXRD system is Bruker D8 discover four-circle diffractometer, allowing measure the reflections in grazing incidence in-plane diffraction (IP-GID). It equipped with a Cu$K_{\alpha 1}$ source in the focus of a multi-layer x-ray mirror and a Ge(220) hybrid monochromator. XRC of (002), (004), (006) and (102) were recorded without an analyzer for qualitative and quantitative analysis. The horizontal and vertical x-ray beam width can be adjusted by use of adjustable slit placed before the sample. The data collected scope can be adjusted from 0.1°to 3°by an auto-controlled 'antis.slit' placed before the detector. The detailed setup is shown in Fig. 1. XRC of (100) of the samples were measured by IP-GID [5]. All the XRC were recorded in symmetric and skew geometry [9-10].

A Veeco Dimension 3100 atomic force microscopy (AFM) was used in a non-contact mode to determine the as-grown surface dislocations. Cathodoluminescence (CL) investigations were performed in a FEI Quanta 400 FEG scanning electron microscope (SEM) equipped with an Gatan mono-CL3+.

3. RESULTS AND DISCUSSION

When GaN layers grown on sapphire substrate reach several microns, the curvature becomes significant. The radius of curvature r was estimated by measuring the XRC of the (002) reflection at two points separated, following the suggestion by Fewster [15]. The radial-distribution of curvature of a typical sample B along and vertical to <10-10> direction can be induced from Fig. 2. The linear fit can gives the radius of curvature. The linear fits along or vertical to <10-10> direction are good and the *R* along the two direction are almost equal. This indicates that the thickness and stress are uniform, which gives a near-spherical curvature. The curvature *R* is defined as the average along the two directions. The *R* of the four samples is shown in Table 1. It is clearly seen that the samples are highly curved with *R* less than 2.5m.

The FWHM of the XRC depends on factors broadening the reciprocal lattice points (RLPs) in the direction of the scan. When the analyzer is not used, the broadenings are mainly caused by dislocation-induced tilt and twist, wafer curvature and grain size [13]. The experimental results showed that only the horizontal beam width have influence on (00*l*) XRC FWHM, as shown in Fig. 3. Horizontal beam restriction does not have quite the same effect for symmetric XRC of a GaN layer, in which the additional broadening factors of grain size and dislocation-induced tilt are present, although the effects of curvature do become less pronounced as the illuminated area decreases. In general, it is more important to restrict the horizontal beam width to the beam when analyzing low dislocation density samples, as wafer curvature proportionally contributes more to the total rocking curve width from these samples. Furthermore, it can be seen for Fig. 3. (a) that the FWHM of (006) reflection is consistently larger than (004) reflection. Normally the XRC

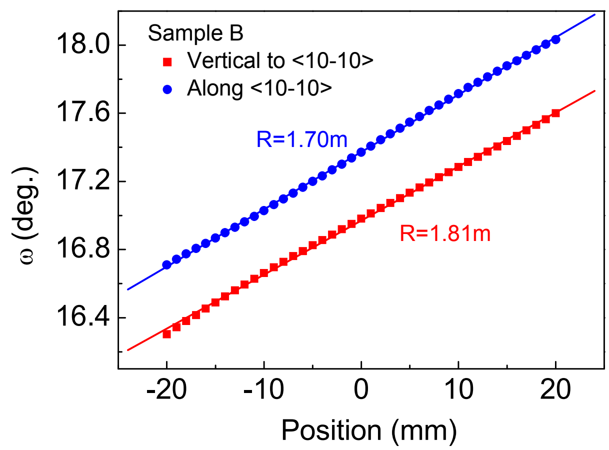

Fig. 2. The peak position of (002) reflection vs. the site on the wafer. We defined the center of the wafer as '0' point.

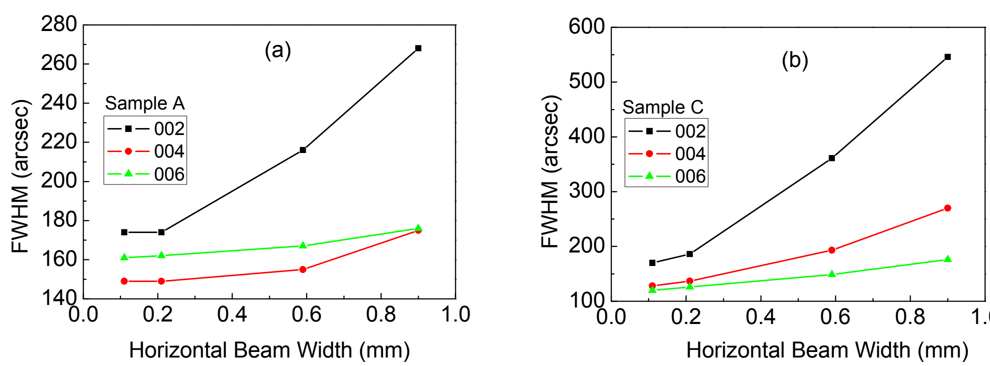

Fig. 3. Influence of horizontal beam width on FWHM of three different symmetric GaN reflections for sample A (a) and C (b).

broadening duo to grain size is expected to decrease as the distance from the RLP to the origin increase [13]. So the (006) reflection should broader than (004) reflection, which should broader than (002) reflection. The three FWHMs of the two 200nm and 600nm MOCVD grown samples can perfectly accord with the expected trend. Howerer, for highly curved wafers, an additional (opposite) trend is present: the high curvature combined with a relatively large illuminated area will tend to increase the broadening of the (002) reflection (and to a lesser extent, the (004) reflection) compared to that of the (006) reflection. The combination of the two trends results in the (002) reflection being larger than the (006) reflection, which is in turn larger than the 004 reflection for sample A. For sample C (have smaller *R* than A), the (002) FWHM is larger than (004) FWHM, which is larger than (002) reflection. This indicates that the curvature-broadening plays a more important role than grain size effect when the curvature becomes more significant.

Also we have investigated the effects of the 'antis.slit' placed before the detector. The experimental results showed that the 'antis.slit' play a similar role as the horitontal beam width. Howerer, the smallest possible 'antis.slit' (0.1°) has a less effects on minimizing the curvature broadening of the (00*l*) FWHM than the smallest possible horizontal beam width (0.11 mm). Therefore, a more precise dislocation-induced tilt angle should be measured with a small horizontal beam width from (004) or (006) reflection for highly curved GaN layers when an analyzer is not used.

The (102) skew symmetric relection is always used to obtain twist angle fast and easily from the following equation:

$$\beta_\omega^2 = (\beta_t \cos\chi)^2 + (\alpha \sin\chi)^2, \quad (1)$$

where β_ω is the measured (102) XRC-FWHM, β_t is the tilt angle, α is the twist angle and χ is the angle between the (102) plane and the sample surface [11-13, 23]. Moram [12] and Liu [14] have showed that the vertical slit width plays a more important role on the curvature-broadening than horizontal slit width. But they have not investigated the influence of the 'antis.slit' when an analyzer was not used. Fig. 4. shows the influence of 'antis.slit' on the (102) FWHM. As can be seen, a smaller 'antis.slit' gives a smaller FWHM. The smallest possible 'antis.slit' is 0.1°for our equipment.

The curvature has little effect on the diffraction from lattice plane perpendicular to the surface, so the (100) XRC were recorded by IP-GID as a standard of gaining twist angles. The twist angles obtained by formula (1) from (102) FWHM (recorded with the smallest possible vertical slit 1mm, the smallest possible 'antis.slit' 0.1°and a relatively larger horizontal slit width 0.2 mm) are listed in Table 1, as well as the twist angles measured by IP-GID. It is can be seen that the twists obtain from the two methods are agreeable. It indicates that the (102) should be recorded with the smallest possible 'antis.slit' and vertical slit of the equipment, while the horizontal slit should be relatively large to increase the diffracted intensity for highly curved wafers when an analyzer is not used. The TTD density obtained by HRXRD, AFM and CL are all listed in Table. 1. The TTD densities obtained by HRXRD are fairly consistent with that obtained by CL and AFM, which indicates that the method to minimize the curvature effect we suggested is acceptable.

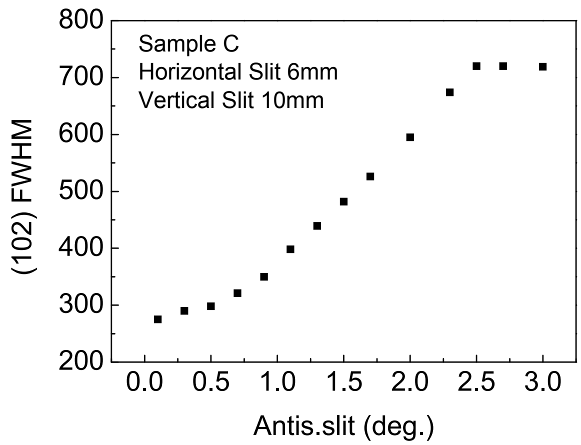

Fig. 4. Influence of 'antis.slit' on the FWHM of skew symmetric (102) reflection of sample C.

Table 1. The detailed message of sample A, B, C and D

Samples	A	B	C	D
Thickness (μm)	12	20	38	300
R (m)	2.46	1.75	1.10	0.45
Twist from this work (arcsec)	385	264	327	170
Twist from IP-GID (arcsec)	397	256	315	152
TTD density from XRD results ($/cm^2$)	7.9×10^8	3.7×10^8	5.7×10^8	1.3×10^8
TTD density from AFM ($/cm^2$)	2.6×10^8	—	2.0×10^8	—
TTD density from CL ($/cm^2$)	2.9×10^8	2.5×10^8	—	0.9×10^8

4. CONCLUSION

In this work we studied the effect of wafer curvature on the XRC of (00*l*) symmetric and (102) skew symmetric reflection when an analyzer was not used. We suggest that a more precise dislocation-induced tilt angle should be measured with the smallest possoble horizontal beam width from (004) or (006) reflection for highly curved GaN layers. While a reliable twist can be obtained from the (102) skew symmetric XRC recorded with the smallest possible vertical slit and 'antis.slit'.

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